# NAREL Standard Operating Procedure
# For Gross Alpha and Beta Analysis of Water Samples

**Effective Date May 16, 2010**

# AM/SOP-4

National Air and Radiation Environmental Laboratory
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## Table of Contents

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## 1.0 PURPOSE

1.1 This method provides a rapid screening measurement to indicate whether specific analyses are required for surface, ground, drinking, and rain water.

## 2.0 SCOPE AND APPLICATION

2.1 This is a screening method to give estimates of the gross alpha activity concentration and gross beta activity concentration in a sample.

2.2 The detection and quantification capabilities of this method are functions of matrix, aliquant size, matrix interferences, mass deposited on the planchet, detection efficiency, background, and counting time. The actual minimum detectable concentration (MDC) for each sample may be different based on any of these variables. For clean water samples, using a 500 mL sample aliquant and a 100 min count, an MDC of 1.8 pCi/L for gross alpha activity and 1.4 pCi/L for gross beta activity are obtainable.

2.3 Since this method does not determine the isotopic composition of the sample, no correction for nuclear decay is possible. All results are referenced to the date and time at the start of the nuclear counting measurement.

2.4 A known volume of a water sample (typically from 10 mL to 1 L) is evaporated to a small volume. The sample is then quantitatively transferred to a planchet. The planchet is dried, weighed, and counted for gross activity.

2.5 Volatile radionuclides such as $^{3}H$, $^{14}C$, and iodine isotopes will not be detected using this method. If flaming is necessary, polonium and cesium may be lost.

2.6 **DISCLAIMER**: Gross screening analyses are intended to provide rapid information associated with a particular action level with minimal chemical preparation. Therefore, these analyses are not as accurate as their isotope-specific counterparts.

2.7 *Interferences*

2.7.1 If the sample residue is hygroscopic, extreme care must be taken to keep the residue dry. Moisture in the source interferes with counting and contributes to self-absorption.

2.7.2 Residue density affects counting and self-absorption of alpha activity measurements. The maximum thickness should be less than 5 mg/cm$^2$ (on 20 cm$^2$ area, 100 mg). The calibration curve extends to a maximum of 7.5 mg/cm$^2$ (150 mg). Under no circumstances may the residue mass exceed the calibration limit of 150 mg.

## 3.0 DEFINITIONS

3.1 **CERLS** – acronym for Center for Environmental Radioanalytical Laboratory Science, formerly the Monitoring and Analytical Services Branch (MASB) – the Center at NAREL that is responsible for analyzing samples for radioactive constituents and hazardous chemicals.

3.2 **control chart** – a graph for monitoring the outputs of a process, such as an analytical measurement process, for the purpose of detecting conditions or trends adverse to quality.

3.3 **crosstalk** – in this method, the misidentification of alpha-particles as beta-particles, or vice versa (sometimes called spillover).

3.4 **laboratory control sample (LCS)** – an artificial sample generated by the analyst in the laboratory and spiked with a known amount of one or more analytes. After being spiked, the LCS is prepared and analyzed in the same manner as a normal sample, and the result of the measurement is compared to the known amount of analyte added to assess the bias of the measurement process.

3.5 **LIMS** – acronym for Laboratory Information Management System – a database and software system used to manage laboratory data, monitor work processes, and produce reports.

3.6 **matrix spike sample (MS)** – an artificial sample generated by the analyst in the laboratory. An aliquant is taken from one sample at the same time another aliquant is taken for normal preparation and analysis. The second aliquant is spiked with a known amount of one or more analytes. After being spiked, the MS is prepared and analyzed in the same manner as a normal sample, and the result of the measurement is compared to the measurement of the unspiked aliquant to assess possible effects of the sample matrix on the analytical results.

3.7 **method blank** – an artificial sample generated by the analyst in the laboratory, which is as free as possible of the analyte of interest. The method blank is prepared and analyzed in the same manner as a normal sample and alongside real samples, so that the result of the measurement may be used to assess low-level bias in the measurement process, such as that caused by contamination of reagents, as well as cross-contamination of samples.

3.8 **MSDS** – acronym for Material Safety Data Sheet, a document that contains information on the potential health effects of exposure to chemicals or other potentially dangerous substances, and on safe working procedures workers should adhere to when handling chemical products.

3.9 **NAREL** – National Air and Radiation Environmental Laboratory.

3.10 **NIST** – acronym for National Institute of Standards and Technology, formerly the National Bureau of Standards (NBS) which is the national standards body for the United States and a member organization of the International Organization for Standardization (ISO).

3.11 **R value** – the ratio of observed activity divided by the actual amount of added activity, a measure of recovery.

3.12 **replicate sample (duplicate)** – an aliquant taken from one sample at the same time another aliquant is taken for normal preparation and analysis. Both aliquants are prepared and analyzed in the same manner. The analytical result for the second aliquant is compared to the result of the first aliquant to assess the precision of the measurement process.

3.13 **SOP** – acronym for Standard Operating Procedure, a document that describes in detail the steps for performing a routine task.

3.14 **self-absorption** – a phenomenon in which some or all of the energy of an emitted ray or particle is absorbed by the solid matrix before the ray or particle reaches the surface of the solid.

## 4.0 EQUIPMENT AND SUPPLIES

4.1 Hot plate

4.2 Drying lamp

4.3 Stainless steel planchets, 5 cm diameter, 0.6 cm height, cupped, with concentric rings.

4.4 Pleated filter paper, 24 cm diameter, Whatman 2V.

4.5 Pipets, 1 mL and 2 mL, volumetric or Eppendorf

4.6 Plastic beaker, 5 mL, disposable.

4.7 Plastic beaker, 50 mL.

4.8 Drying oven.

4.9 Low-background gas proportional counter.

4.10 Vacuum desiccator.

4.11 Bunsen burner.

4.12 Analytical balance

4.13 Assorted glassware

## 5.0 REAGENTS AND STANDARDS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available.

5.2 Sodium carbonate ($Na_2CO_3$), anhydrous, [CAS: 497-19-8].

5.3 Sodium carbonate ($Na_2CO_3$), 100 mg/mL. Dissolve 25.0 g of $Na_2CO_3$ in 250 mL of deionized water.

5.4 Ethyl alcohol ($C_2H_3OH$), [CAS: 64-17-5]. Reagent grade.

5.5 Nitric acid, ($HNO_3$), 16 M, [CAS: 7697-37-2]. Reagent grade.

5.6 Nitric acid, ($HNO_3$), 8 M. Dilute 500 mL of 16 M $HNO_3$ to 1 L with demineralized water.

5.7 Nitric acid, ($HNO_3$), 1 M. Dilute 62.5 mL of 16 M $HNO_3$ to 1 L with demineralized water.

5.8 Silica gel desiccant, [CAS: 63231-67-4].

5.9 Liquid laboratory detergent.

5.10 Calibrated $^{230}Th$ standard solution, an alpha emitter. (Other alpha emitters, such as $^{241}Am$, may be used if $^{230}Th$ is not available in sufficient quantities.)

5.11 Calibrated $^{137}Cs$ standard solution, a beta emitter.

## 6.0 SAFETY

6.1 All procedures performed at NAREL must be conducted following the requirements detailed in the *NAREL Chemical Hygiene Plan* and the *NAREL Radiation Safety Manual*. Safety precautions associated with handling of chemical reagents, solutions, and all samples are the primary responsibility of the analyst. Any spills or accidents involving hazardous, corrosive, or toxic material must be immediately resolved.

6.2 All NAREL laboratory personnel are expected to use good laboratory practices. Most of the safety training is provided by the SHEM officer. The analyst is expected to comply with all directives given by the SHEM officer, and must take necessary precautions to prevent exposure or injury to both self and co-workers.

6.3 Unnecessary or prolonged exposure to laboratory chemicals should be avoided.

6.4 Sodium carbonate ($Na_2CO_3$), anhydrous, [CAS: 497-19-8], is harmful if inhaled and may cause respiratory tract irritation. Causes skin irritation and may be harmful if absorbed through skin. A lachrymator, it causes eye irritation. Ingestion may cause irritation of the digestive tract; may be harmful if swallowed.

6.5 Ethyl alcohol ($C_2H_3OH$), [CAS: 64-17-5], is flammable as a liquid and as a vapor. Inhalation may cause drowsiness and irritation to the respiratory tract. Avoid skin and eye contact by using appropriate protective clothing. Use only in a well-ventilated area away from open flames and ignition sources. Store in containers approved for ethyl alcohol.

6.6 Nitric acid, ($HNO_3$), [CAS: 7697-37-2], is poisonous, reactive, and a strong oxidizer. Contact with other materials may cause fire. It can cause burns to body tissues and may be fatal if ingested or inhaled. Vapors are irritating to eyes and mucous membranes. Use only with adequate ventilation and proper protective clothing and gloves. Nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, and combustible organics. Store away from light and heat.

6.7 Silica gel desiccant, [CAS: 63231-67-4], dust is irritating to the respiratory tract if inhaled, and may irritate eyes and skin. Ingestion may cause gastrointestinal irritation with nausea, vomiting and diarrhea.

6.8 Material safety data sheets (MSDSs) are available to all personnel involved in chemical analysis. It is the responsibility of each analyst to be familiar with chemicals used during an analysis.

6.9 Refer to the *NAREL Chemical Hygiene Plan* for verification of appropriate safety and health practices.

## 7.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

7.1 Prior to the collection of an aqueous sample, consideration should be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be followed. Filtration, acid preservation, etc., should be performed at the time of sample collection or as soon thereafter as practically possible. If properly preserved, the sample can be held up to 6 months before analysis.

7.2 Water samples can be shipped to the laboratory and stored in either plastic or glass containers. Nitric acid should be added to the sample in the field to bring the pH to less than 2. Upon receipt of the sample, NAREL staff checks the pH of each water sample for gross alpha/beta analysis and adjusts the pH as necessary.

7.3 For the determination of the dissolved elements, the sample must be filtered through a 0.45 µm pore diameter membrane filter. Acidify the filtrate to pH < 2 using nitric acid immediately following filtration.

7.4 For the determination of total recoverable activity in aqueous samples, acidify with nitric acid at the time of collection to pH < 2. The sample should not be filtered prior to analysis.

7.5 **NOTE**: Following acidification in the laboratory, the sample should be held for 16 hours and the pH verified to be < 2 before withdrawing an aliquant for sample processing.

7.6 Samples for gross alpha/beta analysis do not require refrigeration.

## 8.0 CALIBRATION AND STANDARDIZATION

8.1 For instructions on set up of low background gas proportional counters refer to *NAREL Standard Operating Procedure for Calibration and Use of the Gamma Products G542 and G5400 Alpha/Beta Counting Systems*.

8.2 The NAREL Quality Assurance Manager provides certified NIST-traceable solutions of $^{230}$Th for alpha calibration and $^{137}$Cs for beta calibration.

8.3 Prepare twenty-eight 5 cm diameter stainless steel planchets by washing them with a concentrated liquid laboratory detergent and thoroughly rinsing with demineralized water and ethyl alcohol. (Fourteen planchets are needed for the alpha calibration and fourteen for the beta calibration.)

8.4 Label each planchet with the name of the nuclide and the nominal mass to be added to the planchet. For both alpha and beta calibrations, set up duplicate planchets with the following nominal masses to be added:

10 mg
20 mg
40 mg
70 mg
110 mg
150 mg

Label the duplicates “A” and “B” (e.g., “0A” and “0B”, “10A” and “10B”).

8.5 Weigh each planchet and record the mass empty in the logbook.

8.6 Based on the activity of the calibration solution ($^{230}$Th or $^{137}$Cs) and historical information about the detector counting efficiencies (if available) calculate the amount of activity to add to each planchet. If possible, there should be sufficient activity to provide a count rate of at least 1500 cpm in the appropriate channel (alpha or beta), allowing count times of 10 min or less.

8.7 Label twenty-eight 5 mL disposable beakers to match the labeled planchets.

8.8 Add the appropriate volume of the standard solution into each of the labeled 5 mL beakers.

8.9 A 100 mg/mL solution of sodium carbonate ($Na_2CO_3$) is used to provide the mass for the self-absorption calibration. Using calibrated Eppendorf pipets, pipet increasing aliquants of the $Na_2CO_3$ solution into 5 mL disposable plastic beakers to represent the nonzero residue masses listed in Step 8.4.

| For mass: | Pipet volume: |
|---|---|
| 0 mg | 0 mL |
| 10 mg | 0.10 mL |
| 20 mg | 0.20 mL |
| 40 mg | 0.40 mL |
| 70 mg | 0.70 mL |
| 110 mg | 1.10 mL |
| 150 mg | 1.50 mL |

8.10 After pipetting the appropriate volume of the standard solution from Step 8.6 and sodium carbonate solution from Step 8.9 into each beaker, add decreasing amounts of 1 M $HNO_3$ to each beaker. Final total volume in each beaker should be approximately 4 mL. Mix gently. Prepare alpha and beta standards separately.

**NOTE**: It is important that the standard solution and the absorbing material ($Na_2CO_3$) be mixed to make the calibration sources more representative of actual samples.

8.11 Using a transfer pipet, incrementally add the entire volume of solution from each beaker to the corresponding planchet while drying under a heat lamp. Make every effort to ensure that the solution covers the bottom of the planchet evenly but does not creep up the sides.

8.12 After all the solution has been transferred, rinse the beaker and pipet with two aliquants of 1 mL each of 1 M $HNO_3$, and add the rinse to the planchet.

8.13 Allow the planchets to dry under the heat lamp. Then place the planchets in a drying oven with constant temperature of between 100 and 105 ºC for at least one hour to ensure complete drying.

8.14 Place planchets in vacuum desiccator for at least one hour.

8.15 Weigh each planchet with mass added, and record the mass in the logbook. Subtract the planchet-empty mass from the final mass and record the net value.

8.16 Using the printed calibration forms, document the measured mass for each planchet and the amount of activity added to each. Appendix 17.1 contains a copy of the Gross Alpha Calibration Form. Appendix 17.2 contains a copy of the Gross Beta Calibration Form.

8.17 Count each of the seven $^{230}Th$ sources in each detector long enough to obtain at least 15 000 alpha counts.

8.18 Count each of the seven $^{137}Cs$ sources in each detector long enough to obtain at least 15 000 beta counts.

8.19 Use regression to fit curves to the efficiency-versus-mass data points. Print graphs of the curves.

8.20 Use regression to fit curves to the beta counting data for the alpha calibration sources. Print graphs of the curves. These curves account for crosstalk of alpha counts in the beta counting channels.

8.21 The curve fit for an alpha calibration is acceptable if the root mean square relative error is less than 10 % and no single data point deviates from the curve by more than 20 % of the fitted value. The beta curve fit is acceptable if no single data point deviates from the curve by more than 10 %.

8.22 Prepare a table of the alpha and beta curve parameters for all the detectors.

8.23 Verify the calibration curves by preparing and assaying three samples of known alpha activity and three samples of known beta activity, using certified traceable standards that contain the same nuclides as the standards used for calibration but that have been obtained from different vendors or lots. Each set of three should include samples with residue masses in the range (10 – 20) mg, (60 – 70) mg, and (120 – 130) mg. The activity of each source should be enough to generate at least 2500 net counts in the primary channel (ignoring crosstalk) in the desired count time (typically 100 min or less). Compare the assayed results with the certified results by calculating and evaluating the *Z* score (see equation 20 in 11.12). A calibration curve is considered to be verified if the absolute value of each associated *Z* score does not exceed 3.

## 9.0 PROCEDURE

9.1 If undissolved solids are present in the water sample, filter the water sample through a pleated filter paper, using suction if necessary, and then acidify the sample with $HNO_3$ (2.5 mL of 16 M $HNO_3$ per liter of sample).

9.2 If required, gross alpha and beta activity measurements can be performed for the insoluble fraction. Dry and ash the filter paper at 500 °C for 12 to 24 hours.

9.3 Transfer a specific volume of water from each sample to a labeled beaker and evaporate to a small volume on a hot plate. Avoid dryness.

9.4 Add 2 mL of sample immediately to the planchet, and evaporate under a heat lamp.

9.5 Repeat Step 9.4 until all of the water sample has been transferred to the planchet. Rinse the beaker and transfer the rinsate to the planchet, repeating Step 9.4.

9.6 Dry each sample under a heat lamp.

9.7 Place each sample in a drying oven with constant temperature of between 100 °C and 105 °C for at least 1 hour.

9.8 Cool completely in a vacuum desiccator and weigh the source. Record the gross mass in the logbook and calculate and record the net mass ($m$ in Section 11). Store the sources in a desiccator. The maximum mass on the planchet should be no more than 100 mg.

9.9 If a sample is hygroscopic, flame the sample over a burner until dull red and repeat Step 9.8.

**NOTE**: Flaming the planchet will result in the loss of $^{210}$Po and possibly some $^{137}$Cs if present. A. sample is considered hygroscopic when a constant mass cannot be obtained by placing the planchet on the weighing pan for at least fifteen seconds: ie mass continues to fluctuate.

9.10 Count for gross alpha and gross beta activity.

9.11 If necessary, dissolve or slurry the ash from Step 9.2 in 5 mL of 8 M $HNO_3$, and repeat Steps 9.3 through 9.10 for the ash.

## 10.0 QUALITY CONTROL PROCEDURES

10.1 For each QC batch of up to 20 samples of the same aqueous matrix, the analyst must add the following quality control samples:

10.1.1 method blank

10.1.2 laboratory control sample (LCS)

10.1.2.1 The same calibrated solutions of $^{230}$Th and $^{137}$Cs should be used for spiking both the LCS and the matrix spike samples. (See 10.1.4).

10.1.2.2 The activity of an analyte added to the LCS must be at least five times the normal expected minimum detectable activity (MDA) for that analyte and should be comparable to sample activities when sample activities in a batch are expected to be higher than five times the MDA. The spike level should be high enough to ensure that under expected measurement conditions the relative standard counting uncertainty will not exceed 5 %.

10.1.3 replicate sample (duplicate)

10.1.4 matrix spike sample

10.2 Analysts are required to control chart results from LCS and blank samples, and to observe the control charts for indicators of possible problems in the measurement system. LIMS software allows the analyst to input data points and to view and print the control charts.

10.3 See the *NAREL Radiochemistry Quality Assurance Manual* for acceptance criteria for QC samples, contingencies for failed QC samples, and equations for calculating values for quality indicators.

## 11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Calculate the alpha and beta detection efficiencies, the alpha-channel detection efficiency for beta emissions, and the beta-channel detection efficiency for alpha emissions as follows.

$$\varepsilon_{\alpha\alpha} = \begin{cases} \varepsilon_{\alpha\alpha,0} \times \left(1 - \dfrac{\nu_\alpha}{\nu_\alpha + 1}\left(\dfrac{m}{R}\right)^{\mu_\alpha}\right), & \text{if } m \le R \\ \varepsilon_{\alpha\alpha,0} \times \dfrac{(m/R)^{-\mu_\alpha \nu_\alpha}}{\nu_\alpha + 1}, & \text{if } m > R \end{cases} \quad (1)$$

$$\varepsilon_{\beta\beta} = \varepsilon_{\beta\beta,0} \times \frac{1 - e^{-\mu_\beta m}}{\mu_\beta m} \quad (2)$$

$$\varepsilon_{\alpha\beta} = \left(\varepsilon_{\alpha\beta,0} \times \frac{1 - e^{-\mu_{\alpha\beta} m}}{\mu_{\alpha\beta} m}\right) + C_{\alpha\beta} \quad (3)$$

$$\varepsilon_{\beta\alpha} = 0 \quad (4)$$

where

| | |
|---|---|
| $\varepsilon_{\alpha\alpha}$ | is the alpha detection efficiency, |
| $\varepsilon_{\beta\beta}$ | is the beta detection efficiency, |
| $\varepsilon_{\beta\alpha}$ | is the alpha-channel detection efficiency for beta emissions, |
| $\varepsilon_{\alpha\beta}$ | is the beta-channel detection efficiency for alpha emissions, |
| $m$ | is the sample residue mass (mg), |
| $\varepsilon_{\alpha\alpha,0}$ | is the zero-mass alpha detection efficiency (a calibration parameter), |
| $\varepsilon_{\beta\beta,0}$ | is the zero-mass beta detection efficiency (a calibration parameter), |
| $\varepsilon_{\alpha\beta,0}$ | is the zero-mass beta-channel detection efficiency for alpha emissions (a calibration parameter), |
| $R$ | is (roughly) the alpha "infinite thickness" in milligrams (a calibration parameter), |
| $\mu_\alpha, \nu_\alpha$ | are shape parameters for the planchet (the value of one of these parameters is fixed at 1 and the other is determined by regression), |
| $\mu_\beta$ | is the beta absorption parameter (a calibration parameter), |
| $\mu_{\alpha\beta}$ | is the alpha-to-beta absorption parameter (a calibration parameter), and |
| $C_{\alpha\beta}$ | is an empirical calibration parameter, which allows the alpha-to-beta efficiency curve to decrease more slowly with mass. |

**NOTE 1**: The alpha-channel detection efficiency for beta emissions (beta-to-alpha crosstalk) is assumed to be negligible, but the equations below are written so that it may be included if necessary.

**NOTE 2**: It is permissible to use cubic polynomials instead of the equations shown above for all the efficiency curves; however, one must never extrapolate a polynomial curve beyond the calibrated mass range.

**NOTE 3**: The "shape" parameters, $\mu_\alpha$ and $\nu_\alpha$, account for the fact that the bottom surface of the planchet is not flat. If the planchet were flat, both of these parameters should be equal to 1, and in this case the parameter $R$ would correspond to the source's "infinite thickness" for alpha-particles. For the planchets that NAREL actually uses, best results have usually been obtained by fixing $\mu_\alpha$ at 1 and finding $\varepsilon_{\alpha\alpha,0}$, $\nu_\alpha$, and $R$ by regression.

**NOTE 4**: The beta absorption factor, $(1 - e^{-\mu_\beta m})/\mu_\beta m$, in equation 2 equals 1 when $m$ is zero, and may be calculated as $e^{-\mu_\beta m/2} \times \sinh(\mu_\beta m/2)/(\mu_\beta m/2)$ when $m$ is nonzero.

11.2 Calculate the alpha-to-beta crosstalk factor and the beta-to-alpha crosstalk factor as follows:

$$X_{\alpha\beta} = \frac{\varepsilon_{\alpha\beta}}{\varepsilon_{\alpha\alpha}} \quad \text{and} \quad X_{\beta\alpha} = \frac{\varepsilon_{\beta\alpha}}{\varepsilon_{\beta\beta}} \tag{5}$$

where

$X_{\alpha\beta}$ is the alpha-to-beta crosstalk factor, or the ratio of the number of misidentified alpha-particles to the number of correctly identified alpha-particles,
$X_{\beta\alpha}$ is the beta-to-alpha crosstalk factor,
$\varepsilon_{\alpha\alpha}$ is the alpha-particle detection efficiency (in the alpha channel),
$\varepsilon_{\beta\beta}$ is the beta-particle detection efficiency (in the alpha channel),
$\varepsilon_{\beta\alpha}$ is the detection efficiency for beta-particles in the alpha channel, and
$\varepsilon_{\alpha\beta}$ is the detection efficiency for alpha-particles in the beta channel.

**NOTE**: The product of $X_{\alpha\beta}$ and $X_{\beta\alpha}$ must be less than 1.

11.3 Estimate the variances of the crosstalk factors as follows:

$$u^2(X_{\alpha\beta}) = X_{\alpha\beta}^2\left(u_r^2(\varepsilon_{\alpha\beta}) + u_r^2(\varepsilon_{\alpha\alpha})\right) \quad \text{and} \quad u^2(X_{\beta\alpha}) = X_{\beta\alpha}^2\left(u_r^2(\varepsilon_{\beta\alpha}) + u_r^2(\varepsilon_{\beta\beta})\right) \tag{6}$$

where

$u_r(\varepsilon_{\alpha\beta})$ is the relative standard uncertainty of $\varepsilon_{\alpha\beta}$,
$u_r(\varepsilon_{\alpha\alpha})$ is the relative standard uncertainty of $\varepsilon_{\alpha\alpha}$ (assumed to be 0.10, or 10 %),
$u_r(\varepsilon_{\beta\alpha})$ is the relative standard uncertainty of $\varepsilon_{\beta\alpha}$ (assumed to be 0 if $\varepsilon_{\beta\alpha}$ is 0), and
$u_r(\varepsilon_{\beta\beta})$ is the relative standard uncertainty of $\varepsilon_{\beta\beta}$ (assumed to be 0.05, or 5 %).

11.4 Calculate the net alpha count rate and the net beta count rate as follows:

$$R_\alpha = \frac{C_{S\alpha}}{t_S} - \frac{C_{B\alpha}}{t_B} \quad \text{and} \quad R_\beta = \frac{C_{S\beta}}{t_S} - \frac{C_{B\beta}}{t_B} \tag{7}$$

where

$R_\alpha$ is the net count rate in the alpha channel ($\text{min}^{-1}$),
$R_\beta$ is the net count rate in the beta channel ($\text{min}^{-1}$),
$C_{S\alpha}$ is the total sample count in the alpha channel,
$C_{B\alpha}$ is the total background count in the alpha channel,
$C_{S\beta}$ is the total sample count in the beta channel,
$C_{B\beta}$ is the total background count in the beta channel,
$t_S$ is the sample count time (min), and
$t_B$ is the background count time (min).

11.5 Estimate the variances of the net count rates as follows:

$$u^2(R_\alpha) = \frac{C_{S\alpha}+1}{t_S^2} + \frac{C_{B\alpha}+1}{t_B^2} + \xi_{B\alpha}^2 \quad \text{and} \quad u^2(R_\beta) = \frac{C_{S\beta}+1}{t_S^2} + \frac{C_{B\beta}+1}{t_B^2} + \xi_{B\beta}^2 \tag{8}$$

where

$\xi_{B\alpha}$ is the excess (non-Poisson) uncertainty in the alpha background correction (assumed to be 0.05 $\text{min}^{-1}$), and

$\xi_{B\beta}$ is the excess (non-Poisson) uncertainty in the beta background correction (assumed to be 0.05 $min^{-1}$).

11.6 Correct the net count rates for crosstalk as follows:

$$R'_\alpha = \frac{R_\alpha - R_\beta X_{\beta\alpha}}{1 - X_{\alpha\beta} X_{\beta\alpha}} \quad \text{and} \quad R'_\beta = \frac{R_\beta - R_\alpha X_{\alpha\beta}}{1 - X_{\alpha\beta} X_{\beta\alpha}} \tag{9}$$

11.7 Estimate the variances of these corrected count rates as follows:

$$u^2(R'_\alpha) = \frac{u^2(R_\alpha) + X_{\beta\alpha}^2 u^2(R_\beta) + R'^2_\alpha X_{\beta\alpha}^2 u^2(X_{\alpha\beta}) + R'^2_\beta u^2(X_{\beta\alpha})}{(1 - X_{\alpha\beta} X_{\beta\alpha})^2} \tag{10}$$

$$u^2(R'_\beta) = \frac{u^2(R_\beta) + X_{\alpha\beta}^2 u^2(R_\alpha) + R'^2_\beta X_{\alpha\beta}^2 u^2(X_{\beta\alpha}) + R'^2_\alpha u^2(X_{\alpha\beta})}{(1 - X_{\alpha\beta} X_{\beta\alpha})^2} \tag{11}$$

11.8 Calculate the alpha and beta activity concentrations as follows:

$$x_\alpha = \frac{R'_\alpha}{2.22 \times V \times \varepsilon_{\alpha\alpha}} \quad \text{and} \quad x_\beta = \frac{R'_\beta}{2.22 \times V \times \varepsilon_{\beta\beta}} \tag{12}$$

where

$x_\alpha$ is the gross alpha activity concentration (pCi/L),
$x_\beta$ is the gross beta activity concentration (pCi/L), and
$V$ is the volume of the sample aliquant analyzed (L).

11.9 Calculate the standard uncertainties of the results as follows:

$$u(x_\alpha) = \sqrt{\frac{u^2(R'_\alpha)}{2.22^2 \times V^2 \varepsilon_{\alpha\alpha}^2} + x_\alpha^2 \times \left( u_r^2(V) + \frac{1 + X_{\alpha\beta} X_{\beta\alpha}}{1 - X_{\alpha\beta} X_{\beta\alpha}} \times u_r^2(\varepsilon_{\alpha\alpha}) \right)} \tag{13}$$

$$u(x_\beta) = \sqrt{\frac{u^2(R'_\beta)}{2.22^2 \times V^2 \varepsilon_{\beta\beta}^2} + x_\beta^2 \times \left( u_r^2(V) + \frac{1 + X_{\alpha\beta} X_{\beta\alpha}}{1 - X_{\alpha\beta} X_{\beta\alpha}} \times u_r^2(\varepsilon_{\beta\beta}) \right)} \tag{14}$$

where

$u(x_\alpha)$ is the combined standard uncertainty of $x_\alpha$ (pCi/L),
$u(x_\beta)$ is the combined standard uncertainty of $x_\beta$ (pCi/L),
$u_r(V)$ is the relative standard uncertainty of $V$,
$u_r(\varepsilon_{\alpha\alpha})$ is the relative standard uncertainty of $\varepsilon_{\alpha\alpha}$ (assumed to be 0.10, or 10 %),
$u_r(\varepsilon_{\beta\beta})$ is the relative standard uncertainty of $\varepsilon_{\beta\beta}$ (assumed to be 0.05, or 5 %).

11.10 Calculate the critical net activities (decision levels) as follows:

$$x_{\alpha C} = \frac{1.645 \sqrt{\frac{R_{\alpha+\beta} X_{\beta\alpha}}{t_S} + (C_{B\alpha} + X_{\beta\alpha}^2 C_{B\beta}) \frac{t_S + t_B}{t_S t_B^2}}}{2.22 \times V \times \varepsilon_{\alpha\alpha} \times (1 - X_{\alpha\beta} X_{\beta\alpha})} \tag{15}$$

$$x_{\beta C} = \frac{1.645\sqrt{\frac{R_{\alpha+\beta}X_{\alpha\beta}}{t_S} + (C_{B\beta} + X_{\alpha\beta}^2 C_{B\alpha})\frac{t_S + t_B}{t_S t_B^2}}}{2.22 \times V \times \varepsilon_{\beta\beta} \times (1 - X_{\alpha\beta}X_{\beta\alpha})} \quad (16)$$

where

$x_{\alpha C}$ is the critical gross alpha activity concentration (pCi/L),
$x_{\beta C}$ is the critical gross beta activity concentration (pCi/L),

and where

$$R_{\alpha+\beta} = \begin{cases} R_\alpha + R_\beta, & \text{if } R_\alpha + R_\beta \geq 0 \\ 0, & \text{otherwise} \end{cases} \quad (17)$$

A detection decision for gross alpha or gross beta activity can be made when necessary by comparing the measured activity concentration to the corresponding critical activity.

11.11 Calculate the minimum detectable concentrations (MDCs) for alpha and beta as follows:

$$x_{\alpha D} = \frac{2.71\frac{1 + X_{\alpha\beta}X_{\beta\alpha}^2}{t_S(1 - X_{\alpha\beta}X_{\beta\alpha})} + 3.29\sqrt{\frac{x_\beta V \varepsilon_{\beta\alpha}(1 + X_{\beta\alpha})}{t_S} + (R_{B\alpha} + X_{\beta\alpha}^2 R_{B\beta})\frac{t_S + t_B}{t_S t_B}}}{2.22 \times V \times \varepsilon_{\alpha\alpha} \times (1 - X_{\alpha\beta}X_{\beta\alpha})} \quad (18)$$

$$x_{\beta D} = \frac{2.71\frac{1 + X_{\beta\alpha}X_{\alpha\beta}^2}{t_S(1 - X_{\alpha\beta}X_{\beta\alpha})} + 3.29\sqrt{\frac{x_\alpha V \varepsilon_{\alpha\beta}(1 + X_{\alpha\beta})}{t_S} + (R_{B\beta} + X_{\alpha\beta}^2 R_{B\alpha})\frac{t_S + t_B}{t_S t_B}}}{2.22 \times V \times \varepsilon_{\beta\beta} \times (1 - X_{\alpha\beta}X_{\beta\alpha})} \quad (19)$$

where

$x_{\alpha D}$ is the minimum detectable gross alpha activity concentration (pCi/L),
$x_{\beta D}$ is the minimum detectable gross beta activity concentration (pCi/L),
$R_{B\alpha}$ is the background count rate in the alpha channel (cpm),
$R_{B\beta}$ is the background count rate in the beta channel (cpm),
$t_S$ is the sample count time (min),
$t_B$ is the background count time (min),
$\varepsilon_{\alpha\alpha}$ is the alpha-particle detection efficiency,
$\varepsilon_{\beta\beta}$ is the beta-particle detection efficiency,
$X_{\alpha\beta}$ is the alpha-to-beta crosstalk factor,
$X_{\beta\alpha}$ is the beta-to-alpha crosstalk factor, and
$V$ is the sample aliquant volume (L).

11.12 The *Z* score for evaluating calibration verification measurements is calculated as follows:

$$Z = \frac{MV - TV}{\sqrt{u^2(MV) + u^2(TV)}} \quad (20)$$

where

$MV$ = measured value
$TV$ = target value
$u(\cdot)$ = standard uncertainty of a value

The *Z* score is considered acceptable if its absolute value does not exceed 3.

## 12.0 DATA REVIEW

12.1 See the *NAREL Standard Operating Procedure for the Review of Radiochemistry Data* (DR/SOP-2) for general procedures for data review.

12.2 The gas proportional counting system administrator or another person designated by the Nuclear Counting Laboratory Manager performs the first official review of gross alpha and beta analysis results. However, the instrument operator should double-check his or her data entry for each analysis even if he or she is not the designated first reviewer.

12.3 The first reviewer checks the raw instrument data from each analysis and judges the reasonableness of the results. These checks are based on the report printed by NAREL's instrument-control software.

12.4 The reviewer checks the data entry for each analysis by comparing the information printed on the report to the information written by the analyst on the assay batch form.

12.5 The residue mass for a gross alpha/beta analysis of a water sample must not be outside the calibrated range, usually 0 mg – 150 mg. If the mass is outside this range, the reviewer must qualify the results with either the J or the R qualifier.

12.6 Gross beta concentrations in RadNet precipitation samples must be compared to the RadNet action level (currently 16 pCi/L). If the concentration exceeds the action level, the reviewer must submit a RadNet Event Report.

**NOTE**: The in-house review software (see below) for the gross alpha/beta analysis warns the reviewer when the concentration exceeds the action level.

12.7 The reviewer completes the review when he or she runs "AbWin," an in-house software system. The reviewer uses AbWin to calculate results of gross alpha/beta analyses and store them in the LIMS database. The program generates a printout for each completed assay batch, showing raw data and results and the disposition selected by the reviewer (either approval or disapproval).

12.8 When the review is complete, the reviewer initials and dates the AbWin printout. The AbWin printout indicates whether the reviewer approved or disapproved the results for reporting. When results are disapproved, the program requires the reviewer to provide the reason or reasons in the form of a comment, which is stored in the LIMS.

12.9 The Nuclear Counting Laboratory returns the following items to the analyst:

- original assay batch form and any other forms provided by the analyst
- copies of printouts of raw data from the instruments (originals filed in the Nuclear Counting Lab)
- original printout from AbWin, initialed and dated

12.10 The analyst double-checks the data entry performed by the Nuclear Counting Laboratory and also reviews the raw data and results of the analysis. He or she initials and dates the AbWin printout. If the analyst disagrees with the judgment of the first reviewer, he or she must write the reason on the AbWin printout with the initials and date.

12.11 The CERLS Radiochemistry Data Coordinator (RDC) performs an independent final review of the results after they have been stored in the LIMS and the analyst has submitted all documentation. The RDC also initials and dates the software printouts and indicates agreement or disagreement with the judgment of the first reviewer. Differences of opinion must be resolved before the results may be reported.

## 13.0 METHOD PERFORMANCE

13.1 Between May 1999 and July 2006, NAREL participated in several proficiency-testing studies for gross alpha and beta activity in water. The results are summarized below:

| Date | | Target Value/ (pCi/L) | Result 1/ (pCi/L) | Result 2/ (pCi/L) | Result 3/ (pCi/L) | Average (pCi/L) | % R |
|---|---|---|---|---|---|---|---|
| 05/01/1999 | Alpha | 24 | 23.7 | 21.3 | 25.3 | 23.433 | 98 |
| 04/17/2000 | Alpha | 54 | 40.9 | 43.7 | 40.8 | 41.800 | 77 |
| 10/18/2001 | Alpha | 97.5 | 69.2 | 80.7 | 71.7 | 73.867 | 76 |
| 05/22/2002 | Alpha | 22.8 | 23.7 | 24.6 | 21.8 | 23.367 | 102 |
| 02/17/2003 | Alpha | 37.6 | 35.5 | 22.3 | 33.4 | 30.400 | 81 |
| 05/19/2003 | Alpha | 70.3 | 71.6 | 77.8 | | 74.700 | 106 |
| 03/01/2004 | Alpha | 293 | 322 | 300 | 258 | 293.333 | 100 |
| 05/01/2004 | Alpha | 1.24 | 1.41 | | | 1.410 | 114 |
| 01/01/2006 | Alpha | 0.581 | 0.637 | | | 0.637 | 110 |
| 07/01/2006 | Alpha | 1.033 | 0.897 | | | 0.897 | 87 |
| | | | | | | | |
| 05/01/1999 | Beta | 47.75 | 47.8 | 48.6 | 56.9 | 51.100 | 107 |
| 04/17/2000 | Beta | 289 | 280.8 | 276.9 | 272.6 | 276.767 | 96 |
| 07/26/2000 | Beta | 87.5 | 79 | 87.8 | 80.7 | 82.500 | 94 |
| 10/18/2001 | Beta | 192 | 156.4 | 168 | 157.2 | 160.533 | 84 |
| 05/22/2002 | Beta | 189 | 290.4 | 293.1 | 299.1 | 294.200 | 156 |
| 02/17/2003 | Beta | 8.55 | 8.5 | 7.3 | 7.1 | 7.633 | 89 |
| 05/19/2003 | Beta | 363 | 285 | 284.7 | 279.6 | 283.100 | 78 |
| 09/01/2003 | Beta | 1948 | 1397 | 1398 | 1597 | 1464.000 | 75 |
| 03/01/2004 | Beta | 1027 | 1015 | 995 | 1072 | 1027.333 | 100 |
| 05/01/2004 | Beta | 4.07 | 4.47 | | | 4.470 | 110 |
| 03/21/2005 | Beta | 17200 | 14800 | 15700 | 16200 | 15566.667 | 91 |

The average value for % R is 95.1 for Alpha and 108.0 for Beta.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA places pollution prevention as the management option of first choice.

## 15.0 WASTE MANAGEMENT

15.1 The EPA requires that laboratory waste management practices be consistent with all applicable rules and regulations. It is the responsibility of each laboratory to assure adherence to EPA regulations.

15.2 No chemical waste stream is generated by this procedure. However, planchets must be discarded in the radioactive waste receptacle and disposed of in accordance with the *NAREL Chemical Hygiene Plan*.

## 16.0 REFERENCES

16.1 *NAREL Standard Operating Procedure for Document Control,* QA/SOP-1.

16.2 U.S. Department of Health, Education, and Welfare, Public Health Service, "Analysis of Radionuclides in Water," Training Course Manual (1965).

16.3 *Less is Better: Laboratory Chemical Management for Waste Reduction*, American Chemical Society's Department of Government Relations and Science Policy, 1155 16$^{th}$ Street N.W., Washington, D.C. 20036. *The Waste Management Manual for Laboratory Personnel*, American Chemical Society's Department of Government Relations and Science Policy, 1155 16$^{th}$ Street N.W., Washington, D.C. 20036.

16.4 *NAREL Chemical Hygiene Plan.*

16.5 *NAREL Standard Operating Procedure for Calibration, Use, and Maintenance of Pipets* (SE/SOP-4).

16.6 *NAREL Standard Operating Procedure for Calibration and Use of Balances* (SE/SOP-1)

16.7 *NAREL Standard Operating Procedure for Calibration and Use of the Gamma Products G542 and G5400 Alpha/Beta Counting Systems (NC/SOP-6).*

16.8 *NAREL Standard Operating Procedure for the Review of Radiochemistry Data (*DR/SOP-2*).*

16.9 *NAREL Radiochemistry Quality Assurance Manual* (QA/QAM-1)

## 17.0 APPENDICES (TABLES, DIAGRAMS, AND FLOWCHARTS)

17.1 Gross Alpha Calibration

17.2 Gross Beta Calibration

17.3 Flow Chart for Gross Alpha and Beta Analysis of Water Samples

## Appendix 17.1

### Gross Alpha Calibration

Radionuclide: _____ Half-life:_____

Supplier: _____ NAREL ID: _____

Preparer: _____ NAREL Ref. Date _____

Activity: _____ ± _____ (2σ) Unit: dpm/mL

Analyst: _____ Date of Calibration Prep: _____

Planchet type: Ridges Absorber: Sodium Carbonate

Number of sources: 16

| Source ID | Net Wt (mg) | Vol. of Std. Added (mL) | Source ID | Net Wt (mg) | Vol. of Std. Added (mL) |
|---|---|---|---|---|---|
| 0A | | | 70A | | |
| 0B | | | 70B | | |
| 10A | | | 110A | | |
| 10B | | | 110B | | |
| 20A | | | 150A | | |
| 20B | | | 150B | | |
| 40A | | | | | |
| 40B | | | | | |

Counting Date(s): _____

Operator: _____

Remarks: _____

_____

_____

## Appendix 17.2

### Gross Beta Calibration

Radionuclide: _____ Half-life:_____

Supplier: _____ NAREL ID: _____

Preparer: _____ NAREL Ref. Date _____

Activity: _____ ± _____ (2σ) Unit: ___dpm/mL___

Analyst: _____ Date of Calibration Prep: _____

Planchet type: ___Ridges_____ Absorber: ___Sodium Carbonate

Number of sources: ____16_____

| Source ID | Net Wt (mg) | Vol. of Std. Added (mL) | Source ID | Net Wt (mg) | Vol. of Std. Added (mL) |
|---|---|---|---|---|---|
| 0A | | | 70A | | |
| 0B | | | 70B | | |
| 10A | | | 110A | | |
| 10B | | | 110B | | |
| 20A | | | 150A | | |
| 20B | | | 150B | | |
| 40A | | | | | |
| 40B | | | | | |

Counting Date(s): _____

Operator: _____

Remarks: _____

_____

_____

**Appendix 17.3**

**Gross Alpha and Beta Analysis of Water Samples**